IN THE SPECIFICATION:

Please amend paragraph number **[0004]** as follows:

[0004] The performance of CL-20 in propellant and weapon systems is highly dependent upon the crystal polymorph of CL-20. CL-20 may undertake several different crystal polymorphs, the most preferred of which is a high density phase known in the art and referred to herein as the ε-polymorph (or epsilon-polymorph) of CL-20. The ε-polymorph of CL-20 is preferred because of the high energetic performance and density, and lower sensitivity compared to other polymorphs. However, many conventional CL-20 synthesis techniques produce ~~non-epsilon~~ non-epsilon polymorphs, especially α-polymorph, in relatively large amounts. The α-polymorph has a much lower density than the ε-polymorph, and, therefore, is less desirable for use in propellant weapon systems. For these reasons, CL-20 synthesized by many conventional techniques must be dissolved and subjected to re-crystallization in order to increase the yield of the ε-polymorph to acceptable levels.

Please amend paragraph number **[0005]** as follows:

[0005] A CL-20 crystallization technique is disclosed in U.S. Patent No. 5,874,574 to Johnston et al., which describes a process by which CL-20 is precipitated into its epsilon polymorph. According to an aspect of this technique, CL-20 is dissolved in a solution containing a non-chlorinated CL-20 solvent, such as ethyl acetate. The CL-20 solvent solution is dried, and a low density non-chlorinated CL-20 non-solvent is then added to the dry CL-20 solvent phase to cause precipitation of ε-polymorph CL-20 crystals. ~~Non-solvents~~ Non-solvents include aromatics, such as benzene and toluene and the like, and relatively lower carbon number hydrocarbons.

Please amend paragraph number **[0006]** as follows:

[0006] The technique of the Johnston et al. patent is particularly effective over most conventional methods in crystallizing ε-polymorph CL-20 prepared from its TADF

(tetraacetyldiformylhexaazaisowurtzitane) precursor. However, application of the same crystallization technique to CL-20 prepared from its TADA ~~(2,6,8,12-tetraacetyl-2,4,6,8,10,12-hexaazatetracyclo-[5.5.0.0^{5,9}0^{3,11}]-dodecane~~ (2,6,8,12-tetraacetyl-2,4,6,8,10,12-hexaazatetracyclo-[5.5.0.0^{5,9}0^{3,11}]-dodecane or "TADH") precursor has certain drawbacks. In particular, addition of the non-solvent to the dry CL-20 solvent solution causes precipitating CL-20 crystals to stick to the crystallizer (e.g., container, beaker, or ~~tank)~~ tank) in which the crystallization is conducted. In some instances, as much as 10 to 20 weight percent of the CL-20 crystal yield remains stuck to the crystallizer walls. In order to remove the precipitated CL-20 crystals from the crystallizer, the CL-20 crystals are redissolved into solution with a CL-20 solvent, such as ethyl acetate, then are recrystallized with ~~a non-solvent.~~ non-solvent. With each recrystallization, a smaller amount of precipitate sticks to the crystallizer walls. Often, however, this process must be repeated several times to produce a high yield without leaving appreciable amounts of CL-20 stuck to the crystallizer. In addition, the crystals form as unusable large agglomerates because of the inability to grow on all surfaces of the crystal.

Please amend paragraph number **[0007]** as follows:

[0007] Evaporation is another known CL-20 crystallization technique. The evaporation technique involves preparing a saturated solution of solvent and non-solvent, seeding the saturated solution with CL-20 crystal seeds, and evaporating the solvent. The solvent is removed slowly by evaporation, leaving CL-20 crystals in the non-solvent. A drawback to the evaporation technique is its expense and difficulties involved with lot-to-lot (or ~~batch-to-batch)~~ batch-to-batch) reproducibility. Variance in CL-20 particle size distribution and quality from batch to batch often demands the practice of post-crystallization grinding. However, grinding adds to production costs. Also, grinding of energetic materials may raise safety rated risks.

Please amend paragraph number **[0012]** as follows:

[0012] In accordance with the principles of this invention, ~~2,4,6,8,10,12-hexanitro-2,4,6,8,10,12-hexaazatetracyclo [5.5.0.0^{5,9}0^{3,11}] dodecane~~ <u>2,4,6,8,10,12-hexanitro-2,4,6,8,10,12-hexaazatetracyclo[5.5.0.0^{5,9}0^{3,11}]-dodecane</u> (CL-20 or HNIW) is crystallized to its ε-polymorph by a novel method. According to a first aspect, the crystallization method comprises preparing a substantially dry CL-20 solvent solution containing an amount of CL-20 dissolved in a CL-20 solvent. The substantially dry solvent solution is added to a crystallizer containing a CL-20 non-solvent to cause precipitation of epsilon polymorph CL-20 crystals by an inverse precipitate technique. The precipitated epsilon polymorph CL-20 crystals are separated from the ~~non-solvent~~ <u>non-solvent</u> and the solvent.

Please amend paragraph number **[0013]** as follows:

[0013] In accordance with a second aspect of the invention, a method is provided that comprises dissolving an amount of CL-20 into a solution containing a CL-20 solvent and water to form an aqueous phase and a wet ~~CL-20~~ <u>CL-20</u> solvent phase, wherein the CL-20 is dissolved in the wet CL-20 solvent phase. The wet CL-20 solvent solution is substantially dried to thereby form a substantially dry solvent solution containing the CL-20. A base is added to the CL-20 solvent phase to neutralize acidic species. The neutralized, substantially dry solvent solution is added to a crystallizer containing a CL-20 non-solvent to cause precipitation of epsilon polymorph CL-20 crystals by an inverse precipitation technique. The precipitated epsilon polymorph CL-20 crystals may be separated from the non-solvent and the solvent.

Please amend paragraph number **[0014]** as follows:

[0014] In accordance with a third aspect of the invention, there is provided a method for crystallizing epsilon-polymorph CL-20 comprising preparing a substantially dry CL-20 solvent solution containing an amount of CL-20 dissolved in a CL-20 solvent.

A crystallizer containing a CL-20 ~~non-solvent~~ non-solvent and seed crystals of epsilon polymorph CL-20 is provided, and the substantially dry solvent solution is added to the crystallizer containing the CL-20 non-solvent and the seed crystals to cause precipitation of epsilon polymorph CL-20 crystals by an inverse precipitation technique. The precipitated epsilon polymorph CL-20 crystals may be separated from the ~~non-solvent~~ non-solvent and the solvent.

Please amend paragraph number **[0015]** as follows:

[0015] In accordance with an embodiment of each of the above aspects of the invention, the sequence of adding the substantially dry CL-20 solvent solution to the ~~non-solvent (also~~ non-solvent (also known as inverse precipitation) substantially reduces or eliminates the sticking of precipitated ~~epsilon polymorph~~ epsilon-polymorph CL-20 crystals to the wall or walls of the crystallizer in which the inverse precipitation technique is carried out. Also within an embodiment of each of the above aspects of the invention, the resulting CL-20 particles have a relatively narrow particle size distribution. For example but not necessarily by limitation, the precipitated epsilon polymorph CL-20 crystals may comprise particles having maximum diameters of, on average, about 40 μm to about 70 μm.

Please amend paragraph number **[0018]** as follows:

[0018] It is also within the scope of this invention to add a ~~co-non-solvent~~ co-non-solvent to the wet CL-20 solvent solution or the dry solvent solution. In one variation of the invention, the co-non-solvent comprises at least one member selected from the group consisting of naphthenic oil, paraffinic oil, and poly(propylene glycol). The weight ratio of co-non-solvent to non-solvent may be, for example, in a range of from about 5:95 to about 20:80.

Please amend paragraph number **[0020]** as follows:

[0020] In accordance with another embodiment of each of the above aspects of the invention, the method further comprises preparing the CL-20 from ~~2,6,8,12-tetraacetyl-2,4,6,8,10,12-hexaazatetracyclo [5.5.0.0^{5,9}0^{3,11}] dodecane~~ 2,6,8,12-tetraacetyl-2,4,6,8,10,12-hexaazatetracyclo-[5.5.0.0^{5,9}0^{3,11}]-dodecane (TADA).

Please amend paragraph number **[0030]** as follows:

[0030] The dry CL-20 solution is fed into a crystallizer ~~(such~~ (such as a container) containing CL-20 non-solvent and optionally other materials, such as seed crystals of CL-20. Non-solvents that are suitable for use in the present invention preferably have boiling points that will allow for separation from the solvent by distillation. Although not necessarily by limitation, the ~~non-solvents~~ non-solvents preferably have poor CL-20 solubility of not more than 5% weight/volume (g/ml), more preferably not more than 1% weight/volume (g/ml), of CL-20 in the non-solvent. Representative non-solvents include the following: alkanes, such as heptane, hexane, and octane; alicyclic alkanes, such as cycloheptane; arenes, such as benzene, toluene, and xylene; and halogenated hydrocarbons, such as chloroform, 1,2-dichloroethane, and bromobenzene. Certain formates and acetates may also be used. Examples of aryl formates include, by way of example only, phenyl formate, phenalkyl formates, such as benzyl formate and phenethylformate; and benzoyl formates, such as 1-methylpropyl benzoyl formate. The aryl formate can also contain substituents, such as in the case of 4-methoxy benzyl formate, multiple formate moieties, and/or heteroatoms. Non-aromatic formates, such as alkylformate (e.g., heptylformate), ethylene glycol diformate, triethylene glycol diformate, and diethylene glycol diformate, can also be selected as the ~~non-solvent.~~ non-solvent. Examples of aryl acetates include, by way of example, phenyl acetate; phenalkyl acetates, such as benzyl acetate and phenethyl acetate; and benzoyl acetates, such as 1-methylpropyl benzoyl acetate. The aryl acetate can also contain substituents, such as in the case of 4-methoxy benzyl acetate, multiple acetate moieties, and/or heteroatoms. Non-aromatic acetates, such as alkylacetates (e.g., heptyl acetate), ethylene glycol diacetate, triethylene glycol diacetate, and

diethylene glycol diacetate, can also be selected as the ~~non-solvent.~~ non-solvent. However, if one of these non-solvents is selected, then the CL-20 solvent will preferably have a different boiling point from the non-solvent to permit separation and recovery of the CL-20 solvent and the non-solvent.

Please amend paragraph number **[0032]** as follows:

[0032] It is particularly preferred that the non-solvent be used in combination with ~~co-non-solvents,~~ co-non-solvents, and in particular naphthenic and/or paraffinic oils. Preferred co-non-solvents include STAN-PLAS 100, ~~STAN-PLAS~~ STAN-PLAS 300, STAN-PLAS 500, STAN-PLAS 1200, SUNPAR 120, and SUNPAR 150, which are examples of refined naphthenic oils and paraffinic oils. Stan-Plas® oils are distributed through Harwick Standard Distribution Corporation. Other useful co-non-solvents are benzyl formate and/or poly(propylene glycol) (PPG). Other co-non-solvents include hydrocarbons, such as hexane, heptane, octane, and higher chain lengths, as well as branched, cyclic, aromatic (e.g., xylene and toluene), and halogenated hydrocarbons. Ethers, especially those having acceptable boiling points for separation from the solvents in ~~post-crystallization~~ post-crystallization operations, can also be used as the co-non-solvent. Preferred ~~co-non-solvents~~ co-non-solvents improve the polymorph and crystal geometry of the resulting ~~CL-20~~ CL-20 particles. Preferably, the co-non-solvent is present in a weight ratio of ~~co-non-solvent~~ co-non-solvent to non-solvent of from about 5:95 to about 20:80. The ~~co-non-solvent~~ co-non-solvent amount is preferably determined by the concentration that will produce the highest yield while maintaining acceptable monocrystalline geometry. It is within the scope of the invention to use the ~~co-non-solvent~~ co-non-solvent as the exclusive non-solvent.

Please amend paragraph number **[0034]** as follows:

[0034] In a preferred embodiment of this invention, ε-polymorph ~~CL-20~~ CL-20 seed crystals are included in the crystallizer with the non-solvent. The ~~CL-20~~ CL-20 crystal seeds are preferably not more than about 200 μm in

diameter, more preferably about 2 μm in diameter, but are not necessarily limited to these sizes. To obtain CL-20 crystal seeds in this range, CL-20 crystals can be ground or milled by techniques known in the art, such as a fluid energy mill or a ball mill. The quantity of CL-20 crystal seeds to be added to the CL-20 ~~non-solvent~~ non-solvent depends upon the desired crystal sizes of the crystals to be grown. An example of an effective range of CL-20 crystal size is, for example, from above 1 μm to about 50 μm diameter crystals. It should be understood, however, that the process may be tailored to produce smaller or larger size crystals. For example, it may be possible to produce crystal sizes of, on average, approximately 0.2 μm, by such techniques as selecting appropriate size crystal seeds and the concentration of CL-20 in the solvent.

Please amend paragraph number **[0039]** as follows:

[0039] The crystallization method of the preferred embodiments discussed herein can be practiced on CL-20 made from various precursors, but is especially useful for crystallizing CL-20 made by nitration of ~~2,6,8,12-tetraacetyl-2,4,6,8,10,12-hexaazatetracyclo-[5.5.0.05,903,11]-dodecane~~ 2,6,8,12-tetraacetyl-2,4,6,8,10,12-hexaazatetracyclo-[5.5.0.05,903,11]-dodecane ("TADH" or "TADA"). The nitration of TADA is known in the art, such as described in EP 0 753 519. Nitration may be carried out in a mixed acid comprising nitric acid and a strong acid. Although sulfuric acid is preferred for use in combination with the nitric acid, nitric acid can be used alone. According to one example of a method of nitrating TADA, nitration can be performed, for example, at 85° C, with the weight ratio of nitric acid to sulfuric acid HNO_3:H_2SO_4 in a range of from about 6:4 to about 8:2, more preferably about 7:3. The ratio of mixed acid (in milliliters) to TADA (in grams) can be in a range of from about 3:1 to about 30:1, more preferably is about 4:1 to about 8:1, and most preferably is about 6:1. The acid mixture may contain up to about 8% by weight of water, but most preferably is substantially free of water, meaning that it has less than

about 2.5% by weight of water. More preferably, the acid mixture has less than 1% by weight of water.

Please amend paragraph number **[0040]** as follows:

[0040] TADA is available through Asahi of Osaka, Japan. TADA can also be prepared from hexabenzylhexaazaisowurtzitane (HBIW) as a precursor. Preparation of TADH from HBIW is described, for example, in EP 0 753 519. HBIW can be synthesized according to the procedure described by Nielsen et al. in "Polyazapolycyclics by Condensation of Aldehydes with Amines. 2. Formation of ~~2,4,6,8,10,12-Hexabenzyl-2,4,6,8,10,12-hexaazatetracyclo [5.5.0.0^{5,9}.0^{3,11}]-dodecanes~~ 2,4,6,8,10,12-Hexabenzyl-2,4,6,8,10,12-hexaazatetracyclo[5.5.0.0^{5,9}.0^{3,11}]-dodecanes from Glyoxal and Benzylamines," Journal of Organic Chemistry, Vol. 55, pp. 1459-66 (1990) and U.S. Patent No. 5,723,604.